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I also certify that the application is now proceeding in the name as identified herein.

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Signed Stephen Hordley

Dated 22 October 2003

GB 0222331.1

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of

APPLIED MULTILAYERS LIMITED,
290 Hartlebury Trading Estate,
HARTLEBURY,
Worcs,
DY10 4JB,
United Kingdom

Incorporated in the United Kingdom,

[ADP No. 08681769001]

Patents Form 1/77

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265EP02 E751157-1 D02846
P01/7700 0.00-0222331.1

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1.	Your reference	**GW-G33025**
2.	Patent application number *(The Patent Office will fill in this part)*	0222331.1 26 SEP 2002 24/7/0
3.	Full name, address and postcode of the or of each applicant *(underline all surnames)*	**Teer Coatings Limited** **290 Hartlebury Trading Estate** **Hartlebury** **Worcester** **DY10 4JB**
	Patents ADP number *(if you know it)* 6611321002	
	If the applicant is a corporate body, give the country/state of its incorporation	**England**
4.	Title of the invention	**A Method for Depositing Multilayer Coatings with Controlled Thickness**
5.	Name of your agent *(if you have one)*	**Bailey Walsh & Co.**
	"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*	**5, York Place** **Leeds** **LS1 2SD**
	Patents ADP number *(if you know it)*	**224001**

SECTION 30 (1977) APPLICATION FILED

		Country	Priority application number *(if you know it)*	Date of filing *(day / month / years)*
6.	If you are declaring priority from one or more earlier patent applications, give the and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number			

		Number of earlier application	Date of filing *(day / month / years)*
7.	If this application is divided or otherwise derived from an earlier UK application, the earlier application		

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer "Yes" if:* — **Yes**

a) any applicant named in part 3 is not an inventor, or

b) there is an inventor who is not named as an applicant, or

c) any named applicant is a corporate body

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Continuation sheets of this form	
Description	11
Claim(s)	CF
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Drawing(s)	2 + 2

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Priority Documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(Please specify)*	

I/We request the grant of a patent on the basis of this application

Signature [signature] Date 25.09.02

Name and daytime telephone number of person to contact in the United Kingdom	**G Wood** **0113 2433824**

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A Method for Depositing Multilayer Coatings with Controlled Thickness

The invention to which this application relates is to the provision of a method for the deposition of multilayered coatings, in terms of layers of different materials or different material compositions, onto a substrate with the coating and/or the layers in the coating having controlled thickness characteristics.

The method utilised in this invention is based on sputtering and preferably magnetron sputter cathodes, and yet further, unbalanced magnetrons used in accordance with apparatus in the applicant's patent GB2258343. Conventionally, in a typical coating system with two magnetrons which face each other, the first magnetron is provided with a target of material A to be deposited on the substrate and the second magnetron is provided with a target of material B also to be deposited onto the substrate. A power supply is provided for each of the magnetrons on the controlled conditions from any suitable power supply so that the rate of deposition from the magnetron is constant and controlled. The substrates that are mounted on a central rotating holder, receive a coating flux from the target so that material A and material B are alternatively applied to the substrate in layers to form a multilayer coating. The thickness of the individual layers of material is typically defined by the power applied to the magnetrons and by the speed of rotation of the holder of the substrates.

A common problem with this method of application is that the interface between the respective layers in the coating is not well defined or sharp.

It has previously been proposed to solve this problem by providing the magnetrons in a manner so that they can be switched on and off to give alternative levels of control. An adaptation of this process is to provide shutters which can be operated to provide additional control.

The provision of shutters and/or switching on and off of magnetrons can provide difficulties in the generation and operation of control systems. This is particularly problematic for the deposition of coatings for relatively low tolerance uses where imperfection can cause significant problems. An example of this is the application of coatings on optical substrates, where the required degree of control of both composition and thickness is extreme. With the use of conventional shutter arrangements it is not possible to establish stable gas flow in the coating chamber and/or stable target poisoning conditions behind the shutter. As a result the electrical potential, magnetic field and gas flow close to the magnetrons is adversely affected.

The aim of the present invention is to provide a system for the control and application of multilayered coatings onto the substrate which avoids the control and application problems conventionally encountered.

In a first aspect of the invention there is provided apparatus for the application of a multilayered coating, said coating comprising at least first and second types of material or material compositions, said apparatus comprising first and second magnetrons from which materials can be selectively applied, a substrate holder, on which the substrates to be coated are held and characterised in that positioned between the magnetrons and the substrates, is a rotatable shield, said shield including at least one aperture through which material deposited from a magnetron can pass for application on suitably positioned substrates, when the aperture is suitably positioned with respect to the said magnetron.

Typically, the movement of the shield in use means that when the aperture or any part of the aperture is not in front of a magnetron, deposition of the material onto the substrates from that magnetron is prevented.

Typically the shield is cylindrical and is rotated so that the aperture moves from a position in front of a first magnetron to a position in front of the second magnetron and so on, thus allowing, continuous operation of the magnetrons, so that deposition of a layered coating with alternate layers of materials from the first and second magnetron is achieved.

Typically, the thickness of the individual layers of material is controlled by the power applied to the magnetron and the time of the aperture being positioned in front of that particular magnetron. It is found that accurate and close control of thickness of the material layer with improved and sharp interfaces between the layers is achieved by the invention.

Typically the magnetrons can be round or rectangular and the aperture is shaped so as to match the requirements for the particular magnetron used.

Typically, the size and shape of the aperture is chosen to ensure thickness uniformity of the material coating on the substrates.

In one embodiment, the substrates are mounted on a holder which itself is rotating but is typically rotating at a faster rate than the cylindrical shutter.

In a further aspect of the invention there is provided a method for controlling the application of a multilayer coating onto a substrate, said method comprising mounting first and second magnetrons of different materials or material compositions in a coating chamber, mounting the substrates to be coated on a substrate holder in the chamber, interposing between the substrate holder and the magnetrons, a shield, providing in said shield at least one aperture which, when correctly positioned with respect to a magnetron, allows material from the magnetron to pass therethrough and onto the substrates and characterised in that the shield is selectively rotatable so as to move and position the aperture at a position in front of the first or second magnetrons for selected periods of time and then to repeat the same as required with respect to the first and second magnetrons so as to allow the application of a multilayered coating with each said layer comprising material from the first or second magnetron.

Typically, the movement of the shield is continuous at a fixed rate or alternatively, can involve a selected dwell time in front of one or other or both of the magnetrons.

Typically, the shield is selected to be used has a radius so that it can be close to the magnetrons or close to the substrates or any position between these extremes to suit particular coating requirements. Thus a range of shields can be provided to be selectively positioned in the coating chamber to suit particular coating requirements and may be replaced, as required, by alternative shields with different radii and/or aperture dimensions. Alternatively a shield can be provided which allows adjustment to provide suitable dimensions.

Typically, in whichever embodiment the substrate holder is rotatable.

Description of the prior art arrangements and a specific embodiment of the present invention is now described with reference to the accompanying drawings wherein:-

Figure 1 illustrates a conventional, prior art coating system,

Figure 2 illustrates the provision of shutters in a conventional manner;

Figure 3 illustrates one embodiment of the present invention in plan with a cross section along line E-A of the shield of Figures 5;

Figure 4 illustrates a further embodiment of the invention in plan with a cross section along line C-C of the shield shown in Figure 5;

Figure 5 illustrates three shields in different embodiments in accordance with the invention. And

Figure 6 illustrates a further embodiment of the invention in plan with a cross section along lines A-A and B-B of the two shields in Figure 5 to illustrate the aperture in each.

Referring firstly to Figure 1 there is illustrated a coating system with two facing magnetrons, magnetron 1 with the target material A and magnetron 2 with target material B. In operation, power is applied to the magnetrons under controlled conditions so that the substrates 3, which are mounted on a central rotating holder 4, receive a coating flux from targets A and B alternately and are as a result coated by a multilayer coating.

Figure 2 illustrates a known system of the type of Figure 1 which includes a system to attempt to provide improved definition and sharpness between layers in the multilayer coating. The system comprises the provision of magnetrons 1 and 2 which can be switched on and off and also provided with shutters 6 which can be selectively open as shown by shutter 6' and closed, as shown by shutter 6" to provide additional control of the application of the coating material.

In Figure 2 a "double door" shutter arrangement is used although this can be replaced by a single door or a sliding parallel door. What they all have in common is that for the conventional shutter to operate it needs to be close to the magnetron to provide a relatively small, typically the same size as the magnetron target, barrier close to the magnetron. If the conventional shutters were spaced any distance from the magnetron they would not be effective in blocking the coating flux. The present invention provides a solution to this problem.

The deposition system can be simple sputtering in which case the substrates can be at earth potential. Alternatively, sputter ion plating can be used in which case the substrate can be at any negative potential up to -5kv but more typically about -50v. Furthermore the substrate can be provided at a floating potential but in order to ensure stable conditions during the deposition process, the shield can be earthed, floating or biased to a negative potential as required.

With reference to Figure 3, one embodiment of the invention can be used to deposit coatings in the following manner. The shield 10, is cylindrical and is provided with an aperture 12, the aperture being located such that, by rotation of the shield the aperture can be selectively positioned in front of one of the magnetrons 1, 2, and material deposited from that magnetron passes through the aperture 12 and onto the substrates 3 on the holder 4 which rotates. The shield is selectively rotatable so that the aperture 12 can be moved between a position in front of one magnetron, to a position in front of the other magnetron, or indeed yet further magnetrons if provided and then held with the aperture at the selected magnetron for the require time for depositing material from that magnetron onto the substrate.

Thus, with the shield provided the magnetrons can be continually operated but, only when the aperture 12 is located in front of the magnetron, can the material from the magnetron shield reach the substrate.

The deposition method can be used to deposit coatings in a non reactive gas such as argon. The method can also be used for deposition in a reactive mode. If nitrogen gas is introduced then nitride coatings can be deposited, if a hydrocarbon gas is introduced then carbide coatings can be deposited, if oxygen is introduced then oxide coatings can be deposited.

Methods for deposition by reactive magnetron sputtering are well known and will not be described in detail here. The reactive gas flow into the chamber 14 can be controlled by a needle valve or mass flow valve but preferably by a method relying on optical emission monitoring (OEM) with feed back control to a piezo electric valve would be used to ensure close control of the composition of the compound coating.

Using the OEM method it is necessary initially to establish the selected stable conditions for stoichiometric coatings. Movement of conventional shutters close to the magnetron targets can affect the stability of the gas flow and so cause coatings with undesirable compositions to be deposited. Also, movement of this type of shutter can affect the magnetic field and plasma conditions of the magnetron and cause variations in deposition rates.

The shield in accordance with this invention has several advantages. Because of the preferred cylindrical symmetry it affects the stability of the system very little as it rotates. Also it can be positioned at different radii and the optimum radius can be selected so that any effect on stability is minimised as the cylindrical shutter rotates. Also it is easy to bias the cylindrical shutter electrically and the optimum bias for stability can be selected.

When controlling the thickness of layers closely it is necessary to maintain stability at the sputter magnetrons and any shield movement should change the magnetic and plasma environment at the sputter electrodes as little as possible. This is particularly true for magnetron electrodes. During deposition of compound coatings by reactive techniques the deposition conditions are particularly sensitive to changes in the magnetic and plasma environment of the sputter electrode. Also a shutter of the type shown in Figure 2 will influence the flow of gas to the sputter target causing serious instability in the control of coating composition. As the cylindrical shield is symmetrical, it can be sufficiently distant from the sputter electrode to have little effect on the magnetic and plasma environment at the electrode and have little effect on the gas flow conditions close to the electrode. Also, the electrical potential of the shield can be selected to minimise any effects on the potential field of the whole deposition system and the conditions close to the substrates are maintained stable.

It has been found that a particularly successful and simple arrangement is to position the shield between the substrates and unbalanced magnetron sputter electrodes in the closed field arrangement. For most coatings a substrate bias voltage of less than -50v ensures excellent coating quality. Under these conditions the floating potential of the cylindrical shutter is about -20v and good long term stability is maintained in the system as the shutter is moved.

For most compound coatings deposited reactively using the OEM control method the flow can be controlled adequately at one magnetron. The power on the other magnetron can be selected so that it produces stoichiometric coating at the gas flow resulting from the control system at the first electrode. The required layer thicknesses can then be obtained by controlling the dwell time for the aperture in the shield in front of each sputter electrode.

It may be necessary to have separate OEM gas flow control systems at the two electrodes but these can relatively easily be provided.

If the movement of the shield is not sufficiently rapid to give the required sharpness of interface between the layers of the applied coating a second cylindrical shield 20 positioned inside the first shield 10 and moving in the opposite direction 22 to the rotation of the first shield 24 can be added to the system as illustrated in Figure 6. Such an additional shield 20 also has the advantage that the magnetrons 1, 2 can be run with no possibility of deposition on the substrates 3 when the apertures 12, 26 in the two shields 10, 20 are out of line. This is useful during the establishment of stable and required conditions.

The examples given so far are for a two magnetron system. The method is suitable for multi magnetron systems with any number of magnetrons. For a 4 magnetron system a cylindrical shield with a single aperture can be used so that it moves in front of the selected magnetron electrodes in turn. Alternatively the system shown in Fig. 4 can be used. Here magnetrons 1 and 3 are of the same material A and magnetrons 2 and 4 are of material B. Two diametrically opposed apertures 12 are positioned in the shield 10. This method is similar to that for the 2 magnetron system but gives double the deposition rate as the material. A or B can be deposited from two magnetron pairs, 1,3; 2,4, respectively through the two apertures simultaneously.

Figure 5 illustrates three shields which can be selectively used and each is used in one of the embodiments shown in Figures 3, 4 and 6 to some particular purpose. The drives used to rotate and stop the shields can be any suitable powered and controlled drive means.

The use of a cylindrical shield as described above ensures maximum stability and gives excellent control of layer thickness. It has the disadvantage that the flux from one magnetron (or 2 magnetrons in the case of the 4 magnetron system) is wasted and finishes on the surface of the cylindrical shield but this is of less importance than the improvement in stability. However if one or both of the targets was a precious metal such as gold or platinum it would be easy to recover the coating from the surface of the cylindrical shield with close to 100% recovery rate. Even with this disadvantage the improvement achieved in the control of the system parameters and hence the ability to operate the system efficiently and predictably allows the system to be used to apply multilayered coatings only substrates such as optical lens.

It will also be seen from the embodiment of the invention described that the shields can be positioned a significant distance away from the magnetrons in contrast to the conventional system where the shutter is required to be positioned close to the magnetron to be effective. Thus the present invention allows the shield to be associated with the position of the substrates to be coated rather than the magnetron positions. This in turn allows the improved efficiency of the shield with the aperture controlling the area of the application of the material rather than conventional systems where the shutter is required to close the aperture in order to prevent application of material which is frequently not effective.

Diagrams for patent application:
A Method for Depositing Multi Layer Coatings with Controlled Thickness

Figure 1

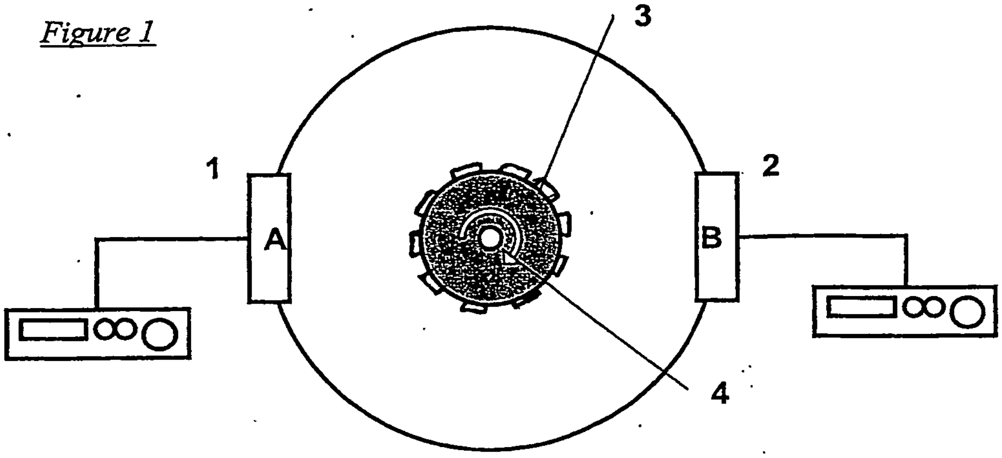

Figure 2

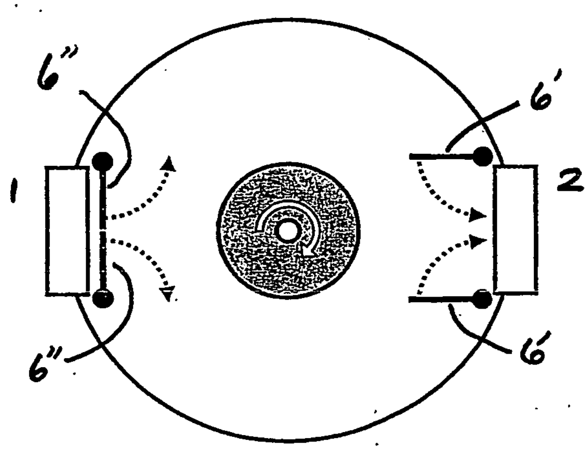

Figure 3

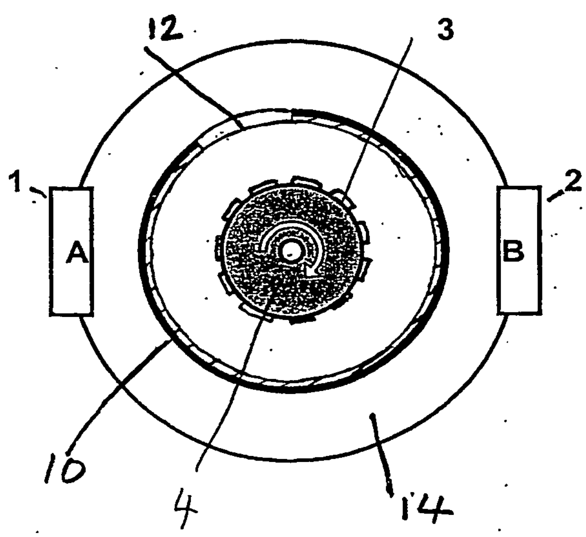

Figure 4

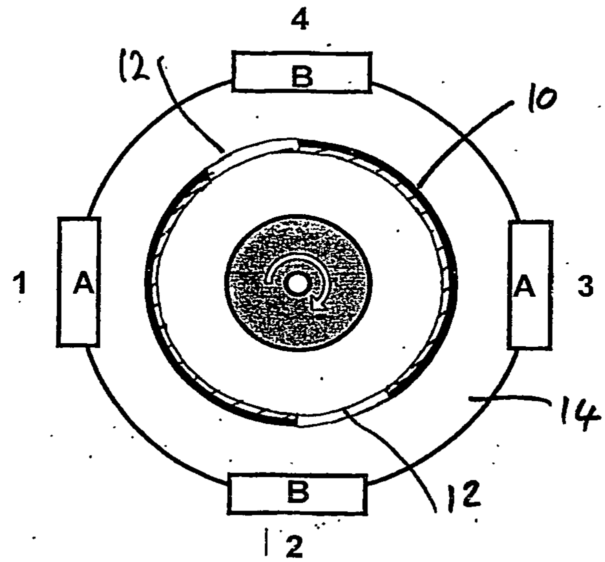

Figure 5

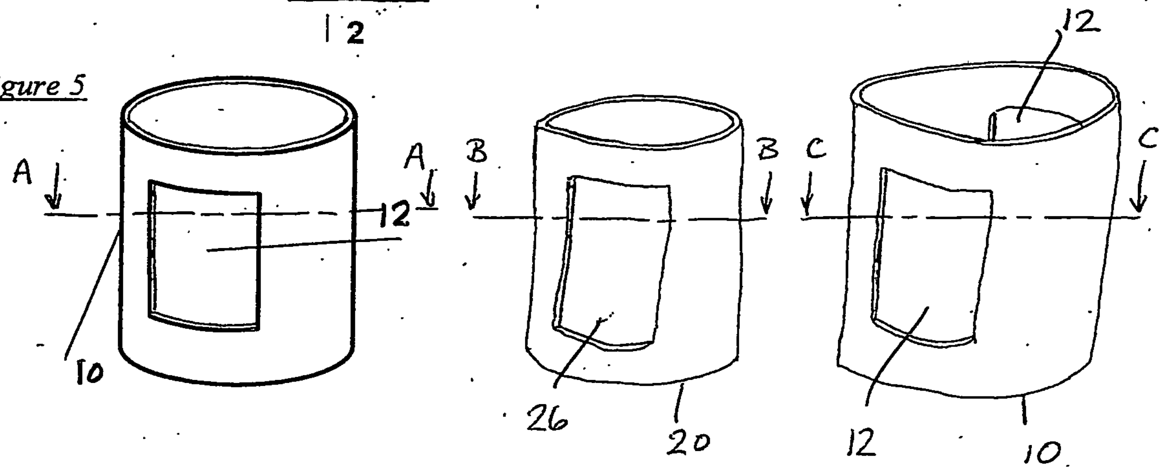

Figure 6